APPENDIX C

QUALITY ASSURANCE PROJECT PLAN

QUALITY ASSURANCE PROJECT PLAN
MISCELLANOUS SITE INVESTIGATIONS
HAMILTON ARMY AIRFIELD

NOVATO, CALIFORNIA

Final Submittal

Prepared by:

US Army Corps
of Engineers ®

Sacramento District
Environmental Design Section

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LIST OF ACRONYMS AND ABBREVIATIONS

ADR	Automated Data Review
bgs	below ground surface
BRAC	Base Realignment and Closure
CCB	Continuing Calibration Blank
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CDQAR	Chemical Data Quality Assessment Report
CESPK	Corps of Engineers, Sacramento District
CL	Control Limit
COC	Chain of Custody
CRWQCB	California Regional Water Quality Control Board
CVAA	Cold Vapor Atomic Absorption
cy	cubic yards
DL	Detection Limit
DoD	Department of Defense
DQO	Data Quality Objectives
DTSC	California Department of Toxic Substances Control
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
ELCD	Electrolytic Conductivity Detector
EPA	Environmental Protection Agency
FITB	Firing-in Target Butt
FSP	Field Sampling Plan
GC	Gas Chromatograph
HAAF	Hamilton Army Airfield
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma (Spectroscopy)

ICS	Interference Check Standard
ICV	Initial Calibration Verification
IS	Internal Standard
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MDL	Method Detection Limit
MS	Matrix Spike
MSA	Method of Standard Addition
MSD	Matrix Spike Duplicate
µg/kg	micrograms per kilogram
mg/kg	milligrams per kilogram
NELAC	National Environmental Laboratory Accreditation Conference
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
P.E.	Professional Engineer
PM	Project Manager
PAHs	Polynuclear Aromatic Hydrocarbons
QL	Quantitation Limit
QA	Quality Assurance
QAC	Quality Assurance Chemist
QAPP	Quality Assurance Project Plan
QC	Quality Control
RF	Response Factor
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
RT	Retention Time
SD	Serial Dilution
SIM	Selective Ion Monitoring
SOPs	Standard Operating Procedures
SPCC	System Performance Check Compounds
Total DDTs	Sum of DDD, DDE, and DDT concentrations

USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
WP	Work Plan

QUALITY ASSURANCE PROJECT PLAN
MISCELLANEOUS SITE INVESTIGATIONS
HAMILTON ARMY AIRFIELD

1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents functions, procedures, and specific quality assurance (QA) and quality control (QC) activities designed to achieve the data quality goals for the various objectives of the sampling efforts at miscellaneous in-board sites described in the Data Quality Objectives (DQOs) at Hamilton Army Airfield. This project is conducted by the Environmental Design Section of the U.S. Army Corps of Engineers, Sacramento District (CESPK) on behalf of the Army Base Realignment and Closure (BRAC) environmental office. This QAPP is prepared in accordance with EPA QA/R-5, EPA Requirements for Quality Assurance Project Plans (U.S. EPA, 2001). This document accompanies the Work Plan (WP), DQOs, and the Field Sampling Plan (FSP).

1.1 Site Location and Project Objectives

The site locations are illustrated in Figure 1-1 of the Work Plan. The objectives for the following sites included in this sampling effort are summarized below.

Spoils Pile F – Confirm full removal of Total DDT-contaminated soil
South of the Runway DDT hotspot – Determine extent of Total DDT hotspot
Building 35 – Determine extent of Total DDT contamination under discharge pipe
Unlined PDD – Determine extent of Total DDTs in the northwest corner of the PDD
Revetments 6&7 – Determine if mercury within these excavations exceeds action goals
Firing-in Target Butt (FITB) – Identify any residual contamination from Department of Defense (DoD) activities
Skeet Range - Identify any residual contamination from DoD activities
Testing Area – Identify any residual contamination from DoD activities

1.2 QAPP Objectives and Use

Standard procedures and specifications are established to ensure that all laboratories produce comparable data, and that data quality is consistently assessed and documented. The specific objectives of this QAPP are to:

- provide standardized references and quality specifications for all anticipated field sampling, analysis, and data review procedures required for the project sites;
- provide guidance and criteria for selected field and analytical procedures; and

- establish procedures for reviewing and documenting compliance with field and analytical procedures.

The fieldwork will include sediment and soil sample collection, field analysis, sample packaging, and shipping to offsite laboratory for analysis.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 Corps of Engineers

The following Sacramento District, Corps of Engineers personnel have been assigned to accomplish the sampling design and execution required supporting this project. The USACE Project Manager is Ray Zimny. The project execution will be performed under the general supervision of Rick Meagher P.E., Chief of Environmental Design Section. The technical team consists of the following personnel:

Technical Team Leader/Chemist:	Kathy Siebenmann	(916) 557-7180
Sampling Team Leader/Geologist:	Teresa Rodgers	(916) 557-6624
Health & Safety Manager:	Donna Maxey	(916) 557-7437
USACE fax number:		(916) 557-7465

2.2 Project Management

2.2.1 Technical Team Leader

The Technical Team Leader will be responsible for reviewing the sampling plans and associated field activities, and ensuring that all sampling activities conform to the QAPP. The Project Leader will oversee quality assurance of field activities. Prior to the start of field activities, preparatory meetings will be held with the field crew. If field conditions require modifications to protocol outlined in the SAP or if questions arise, the Sampling Team Leader or field crew will contact the Technical Team Leader for direction. The Technical Team Leader will also be responsible for overseeing the project and subcontractors, directing field crews, and the compilation of data. The Technical Team Leader reports to the Section Chief.

2.2.2 Project Chemist

The Project Chemist will have a “hands on” role in management of project tasks associated with sampling and analysis. These tasks include:

- Coordination with the analytical laboratory to ensure readiness to implement project specific requirements,
- Review of analytical data as it becomes available to ensure conformance with quality standards, and

- Implementation of corrective actions in accordance with QAPP specifications when review of data uncovers deficiencies.

2.2.3 Sampling Team Leader

The Sampling Team Leader will be responsible for quality assurance of field activities and for executing all work elements related to the sampling program, including documenting field activities, maintaining field notes and photographs, maintaining a record of onsite personnel and visitors, and implementing the sampling plan. These tasks include instruction of field personnel in sampling and preservation requirements and general oversight of field personnel involved in sampling activities.

2.2.4 Health and Safety Manager

The certified industrial hygienist is responsible for the general health and safety plan development and training for field personnel. This individual is also responsible for ensuring that health and safety procedures are understood and followed by all field personnel, and for reporting and correcting any violations of policy or regulations.

2.2.5 Field Crew

Field crew personnel will be responsible for performance of project mobilization, demobilization, sample collection and oversight. Field personnel will report to the Sampling Team Leader. Field personnel will include members of the USACE Environmental Engineering Branch, Sacramento District.

3.0 QUALITY OBJECTIVES FOR ENVIRONMENTAL DATA

The term “data quality” refers to the level of uncertainty associated with a particular data set. Data quality associated with environmental measurement is a function of the sampling plan rationale and procedures used to collect the samples, as well as of the analytical methods and instrumentation used in making the measurements. Uncertainty cannot be entirely eliminated from environmental data. However, quality assurance programs effective in measuring uncertainty in data are employed to monitor and control excursions from the desired data quality objectives (DQOs). The DQO process and data needs are specified in Appendix A of the Work Plan. Sources of uncertainty that can be traced to the sampling component are poor sampling plan design, incorrect sample handling, faulty sample transportation, and inconsistent use of standard operating procedures. The most common sources of uncertainty that can be traced to the analytical component of the total measurement system are calibration and contamination.

The purpose of this QAPP is to ensure that the data collected are of known and documented quality and useful for the purposes for which they are intended. The procedures described are designed to obtain data quality indicators for each field procedure and analytical method. Data quality indicators include the PARCC parameters (i.e., Precision, Accuracy, Representativeness, Comparability, and Completeness). To ensure that quality data continues to be produced, systematic checks must show that test results and field procedures remain reproducible and that the analytical methodology is actually measuring the quantity of analytes in each sample.

A laboratory certified by the State of California and validated by the USACE or successfully audited by National Environmental Laboratory Accreditation Conference (NELAC) auditors will generate all laboratory chemical data. Laboratories must have an in-place program for data reduction, validation, and reporting as discussed in Section 7.0. The reliability and credibility of analytical laboratory results can be corroborated by the inclusion of a program of scheduled replicate analyses, analyses of standard or spiked samples, and analysis of split samples with QA laboratories for some projects. Regularly scheduled analyses of known duplicates, standards, and spiked samples are a routine aspect of data reduction, validation, and reporting procedures.

All data that will be collected for this project will be definitive data using EPA procedures and will be usable in identification, characterization, and engineering design. The data obtained will conform to the quality control requirements specified in the following text and the tables accompanying this document.

4.0 SAMPLE ACQUISITION, CUSTODY, MANAGEMENT, AND DECONTAMINATION

Sample acquisition, custody, management, and decontamination procedures are described in the Field Sampling Plan (FSP) (Appendix B).

The samples will be sent to a State of California and USACE certified or NELAC audited laboratory. The USACE certification includes in-depth audits to determine if quality assurance and quality control measures are in place and adequate. These audits are based upon many of the same elements as the NELAC audits. The address and point of contact will be listed below in the final QAPP pending selection of laboratory through the competitive bidding process.

Point of Contact: Jim Carter
EMAX Laboratories, Inc.
1835 W. 205th Street
Torrance, CA 90501
Phone: (310) 618-8889 #105
Fax: (310) 618-0818

5.0 ANALYTICAL METHODS AND CALIBRATION

This section contains brief descriptions of preparation and analytical methods that will be used to analyze soil samples collected for this project. These methods are listed in Table 5-1.

Table 5-1. Summary of Analytical Methods

Analytes	Preparatory	Analytical
Metals (antimony, arsenic, cadmium, chromium, copper, lead, nickel, zinc)	SW3050B	SW6010B
Mercury	Method	SW7471A
DDT, DDE, DDD	SW3550B, SW3630C	SW8081A
Polynuclear Aromatic Hydrocarbons (PAHs)	SW3550B	SW8270C Modified

If during the course of a project, it becomes necessary to apply a different quantitation limit because of changes in instrument capabilities, the Project Chemist will be notified and approval must first be obtained in instances where higher quantitation limits result. Methodology references contain specific QC criteria associated with the particular methods. These specific requirements include calibration and QC samples, and are described in detail within the methods. Daily performance tests and demonstrations of precision and accuracy are required. These calibration and QC samples are listed in Attachment A to this QAPP.

The laboratory methods identified in this document were published by the United States Environmental Protection Agency (U.S. EPA) in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846*, Third Edition (November 1986; Revision 1, July 1992; and Revision 2, November 1992, Update I, August 1993, Update II, September 1994, Update III, 1998). Preservation and holding times for these analytical procedures are presented in Table 5-2. Attachment A summarizes the calibration and the internal quality control procedures; Attachment B lists the quantitation limits and action goals that will be used for this project.

Table 5-2. Preservation and Holding Times

Method	Chemical Preservation	Holding Time	Temperature Preservation
SW8081A and SW4042	None	14 days before extraction, 40 days after extraction	Cool to 4°C
Modified SW8270C	None	14 days before extraction, 40 days after extraction	Cool to 4°C
SW6010B	None	40 days before digestion, 6 months after digestion	None
SW7471A	None	28 days to analysis	Cool to 4°C

5.1 Sample Preparation and Analytical Field Method

Total DDTs will be analyzed according to Method SW4042 in the field using an immunoassay field test kit to direct stepout sampling. A weighed portion of the soil sample is extracted with deionized water and filtered. An aliquot of the extract and an enzyme-DDT conjugate are added to immobilized DDT antibody. The enzyme-DDT conjugate competes with DDT present in the sample for binding to DDT antibody. The enzyme-DDT conjugate bound to the DDT antibody then catalyzes a colorless substrate to a colored product. The concentration range is indicated by comparing the color of the sample to the response produced by a reference reaction. The reference standard concentrations will include both 0.2 mg/kg and a 1 mg/kg of DDT. The manufacturer's instructions are included in Attachment D.

5.2 Sample Preparation and Analytical Methods - Organic

The following sections briefly summarize the sample preparation and analytical methods to be performed for the determination of organic analytes. Various cleanup methods may be used, depending upon the interferences encountered following extraction. Not all potential cleanup methods are included below. The Project Chemist should be advised of any alternative cleanup methods proposed by the laboratory.

5.2.1 Method SW3550B: Sonication Extraction

Method 3550B is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, wastes, and sludges. The sonication process ensures intimate contact of the sample matrix with the extraction solvent. A weighted portion of the solid material is mixed with the anhydrous sodium sulfate, ground to form a free-flowing powder, and then dispersed into the methylene chloride. The extract is separated from the sample by vacuum or gravity filtration, or centrifugation, and then dried with anhydrous sodium sulfate and concentrated to an appropriate volume for analysis.

5.2.2 Method SW3630C: Silica Gel Cleanup

Generally, solid-phase extraction cartridges filled with silica gel are used. Aliquots of sample extract are loaded onto the cartridges that are then eluted with suitable solvents, depending upon the analysis method. The collected fractions are analyzed by the appropriate method.

5.2.3 Method SW3640A: Gel-Permeation Cleanup

The extract is passed through a column containing a hydrophobic gel absorbent. The column is then flushed with clean organic solvents to separate the interferences from the analytes of interest by retention time.

5.2.4 Method SW8081A: Total DDTs

Method SW8081A is used to determine the concentration of DDD, DDE, and DDT on a gas chromatograph (GC). Prior to analysis, the sample is extracted into solution. An aliquot of solution is injected into an open-tubular capillary column, and detected by an electron capture detector (ECD) or electrolytic conductivity detector (ELCD). Any compounds identified tentatively in the primary analysis are confirmed on a second GC column.

5.2.5 Modified Method SW8270C: Polynuclear Aromatic Hydrocarbons by GC/MS Selective Ion Monitoring

Method SW8270C is used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride. Such compounds include polynuclear aromatic hydrocarbons (PAHs). The concentrated extract is injected into a gas chromatograph for separation and detected by mass spectrometry. Mass spectrometry provides a characteristic ion pattern for fragmented target analytes, providing a high level of confidence in compound identification. Compounds are quantitated by comparing the response of a characteristic ion to the average response from a 5-point calibration. The internal standard technique is used for calibration. The instrument will be modified for selective ion monitoring (SIM) to reduce interferences and lower the quantitation and detection limits of PAHs for this project. Aliquot of the extract is injected into a GC/MS that is set up to detect only specific ions found in the PAH analytes.

5.3 Sample Preparation and Analysis Methods - Inorganic

The following sections briefly summarize the sample preparation and analysis methods to be performed for the determination of inorganic analytes.

5.3.1 Method SW3050B: Acid Digestion of Sediments, Sludges, and Soils

This digestion procedure is used for the preparation of solid samples for analysis by inductively coupled plasma/atomic emission spectroscopy (ICP). A mixture of nitric acid, and the material to be analyzed is refluxed in a covered Griffin beaker or equivalent. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. Hydrogen peroxide is then added and the mixture warmed. The digestate is then cooled and brought to a low volume with water. If the digestate contains suspended solids, it must be centrifuged, filtered, or allowed to settle before analysis.

5.3.2 Method SW6010B: Inductively Coupled Plasma-Atomic Emission Spectrometry

ICP determines elements in solution. The sample requires digestion by Method SW3050B for soil prior to analysis.

The method provides a simultaneous or sequential multi-element determination of elements by ICP. Element-emitted light is measured by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic line emission spectra are produced by radio frequency inductively coupled plasma. The spectra are dispersed and photo-multiplier tubes monitor the intensities of the lines. The spectra are the physical property of the element and the intensity is proportional to the concentration of the element in solution.

5.3.3 Method SW7471A: Cold Vapor Atomic Absorption Spectroscopy

Method SW7471A is based on the absorption of radiation at the 253.7 nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Quantitation is accomplished by comparing the absorbance to a five-point calibration curve prepared from standards of known mercury concentration.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

6.1 Calibration Procedures and Frequency

All instruments and equipment used during sample analysis are operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations, as well as criteria set forth in the applicable analytical methods. Personnel properly trained in these procedures will operate, calibrate, and maintain the instruments. Laboratory capabilities will be demonstrated initially for instrument and reagent/standards performance as well as accuracy and precision of analytical methodology.

Calibration of instruments is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established quantitation limits. Each instrument will be calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method presented in Section 5.0. The frequency of calibration and calibration verification and the concentration of calibration standards are determined by the manufacturer's guidelines and the analytical method. Calibration procedures for all instruments are summarized in the method-specific tables in Attachment A. All samples must be bracketed by passing calibration check samples for the majority of methods. Failure to bracket all samples with acceptable calibration checks may result in the reanalysis of affected samples.

6.1.1 Gas Chromatography

The field of chromatography involves a variety of instrumentation and detection systems. While calibration standards and acceptance criteria vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply uniformly. As outlined in EPA SW-846 procedures, each chromatographic system is calibrated prior to performance of analyses using five concentrations by external standard technique for all columns. The lowest calibration standard shall be within a factor of two relative to the QL, and the others corresponding to the expected range of concentrations or defining the working range of the detector. This is done on each chromatographic column and each instrument at the beginning of the contract period and each time a new column is installed. The results are used to determine a calibration curve and response factors for each analyte. Initial calibration consists of determining the working range, establishing limits of detection, and establishing retention time windows. The calibration is checked on a daily basis to ensure that the system remains within specifications. Second column confirmation is required for single compound analytes.

Continuing calibration standards are analyzed to check the instrument response relative to the initial calibration curve at the beginning and end of each analytical run. Calibration checks are also performed for overall system performance and for retention time shifts, as specified in SW-846. Individual and standard mixes are analyzed to establish response factors and absolute retention time. The response factors and retention times are verified throughout the analytical run and at the end of the analytical sequence. Each analyte must be within its retention time window or the analyst shall take corrective action. For GC analyses conducted on this project, the response factor must agree with the factor determined during the initial 5-point calibration within 15% for quantitation analysis utilizing SW-846 methodology.

The instrumental detection limit, the linear range of the instrument, and interference effects must be established for each individual analyte on that particular instrument. The calibration is verified initially prior to sample analysis using an independent second source standard. Calibration verification standards are analyzed after every 10 samples using a midrange calibration check standard and must be within 15% of the expected value.

6.1.2 GC/MS analysis

Each day prior to analysis of samples, the instrument is tuned with bromofluorobenzene for volatile compounds and decafluorotriphenylphosphine for semivolatile compounds or other tuning criteria as specified by the method used. Mass spectral peaks must conform both in mass numbers and relative intensity to method-specified requirements before analyses can proceed.

The instrument is then calibrated for all target compounds. An initial calibration curve is produced to define the working range to establish criteria for identification. All GC/MS instruments are calibrated at five different concentrations for analytes of interest, using the procedures outlined in SW-846. Method system performance check compounds (SPCC's) must show a minimum mean response factor and method calibration check compounds (CCC) must show a relative standard deviation (RSD) less than the method specified standard for the initial calibration to be considered valid. On a daily basis, SPCC's must meet the same criteria relevant for the initial calibration and CCCs must show a minimum percent drift relative to the expected concentration of the CCC to be considered valid. This initial calibration is evaluated on a daily basis to ensure that the system is within calibration. If the daily standard does not meet the established criteria, the system is recalibrated. These procedures will be modified for selective ion monitoring.

Following a successful tune, the initial five-point calibration is verified by a single mid-range concentration standard. The calibration is verified daily prior to sample analysis using an independent second source standard. This initial calibration can be utilized as long as the calibration verification remains valid.

6.1.3 Inductively Coupled Argon Plasma-Atomic Emission Spectrometry (ICPES) Metals

Plasma emission spectrophotometry, also termed inductively coupled argon plasma (ICP) spectrometry, is calibrated daily using either one standard solution and one blank or a four-point calibration (3 levels plus blank). For the single standard calibration, the calibration standard must be within the demonstrated linear range of the instrument. The instrumental detection limit, the linear range of the instrument, and interference effects must be established for each individual analyte on that particular instrument. The linear range is verified at the time of the analysis by analyzing the highest calibration standard as a sample, the results of which must be within ± 5% of its true value. The calibration is verified initially prior to sample analysis using an independent second source standard at a concentration mid-range of the calibration. Continuing calibration checks are analyzed after every 10 samples using a mid-range calibration check standard and must be within ±10% of the expected value. Sensitivity is established at the lower calibration level by analyzing a low level standard at the QL (3 to 5 times the MDL). Calibration blanks are analyzed after all calibration check standards and no analytes may be detected above one-half the QL. An interelement check standard is analyzed at the beginning and end of each analytical run, to verify that interelement and background correction factors have remained constant. Results outside of the established criteria trigger reanalysis of samples.

6.1.4 Atomic Absorption Spectroscopy

The instrument must be calibrated and checked for contamination before each set of samples. An initial calibration (ICAL) consists of a minimum of a blank and three calibration standards. The least concentrated standard will be at a concentration corresponding to the QL. The remaining standards will define the working range of the instrument. A linear regression fit of the calibration data must yield a correlation coefficient must be at least 0.995. Failure to meet these criteria will require recalibration and possible preparation of a new set of standards. Prior to sample analysis, an initial calibration verification (ICV), consisting of a second source standard, and an initial calibration blank (ICB) will be analyzed to verify the quantitation and to detect any contamination. A continuing calibration verification (CCV) at a mid-curve concentration and CCB will be analyzed very 10 samples and at the end of analytical sequence. If the CCV value varies from the predicted concentration by more than + 10% then the analysis must be stopped. The problem must be identified and corrected, and rerun the impacted samples. All samples must be bracketed by calibration standards that meet the stated criteria.

6.2 Standard and Reagent Preparation

A critical element in the generation of quality data is the purity and traceability of the standard solutions and reagents used in the analytical operations. The preparation and maintenance of standards and reagents will be performed per the specified analytical methods presented in Section 5.0. The laboratory shall continually monitor the quality of reagents and standard solutions through a series of well-documented standard operating procedures (SOPs). In general, SOPs for standards preparation should incorporate the following items:

- Documentation and labeling of date received, lot number, date opened, and expiration date;
- Documentation of traceability;
- Preparation, storage, and labeling of stock and working solutions; and
- Establishing and documenting expiration dates and disposal of unusable standards.

Primary reference standards and standard solutions used by the laboratory are to be obtained from the National Institute of Standards and Technology, or other reliable commercial sources to ensure the highest level of purity possible. All standards and standard solutions shall be catalogued to identify the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information included in the specific SOP.

Standard solutions and reagents are validated prior to use. Validation procedures can range from a check for chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time, concentration or source. Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory. Stock and working standards are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration.

6.3 Field Quality Control Checks

Quality control checks in the field will include the collection of field duplicate and temperature blank samples. These QC checks are described in Section 4.2 of the FSP. In addition, the quality control checks associated with the immunoassay field analytical technique also include collection and analysis of duplicate samples, control samples, and reference standards.

6.4 Laboratory Quality Control Procedures

The Project Laboratories will have a QA/QC program that monitors data quality with internal QC checks. Internal QC checks are used to answer two questions:

1) Are laboratory operations in-control, (i.e., operating within acceptable QC guidelines), during data generation?

2) What effect does the sample matrix have on the data being generated?

Laboratory performance QC is based on the use of a standard control matrix to generate precision and accuracy data that are compared, on a daily basis, to control limits. This information, in conjunction with method blank data, is used to assess daily laboratory performance.

The second question is addressed with matrix-specific QC. Matrix-specific QC is based on the use of an actual environmental sample for precision and accuracy determinations and commonly relies on the analysis of matrix spikes, matrix spike duplicates, and surrogate standards. This information, supplemented with field blank results, is used to assess the effect of the matrix and field conditions on analytical data.

Laboratory performance QC will be provided as a standard part of every routine analysis. Matrix-specific QC frequency will be required per the tables in Attachment A. A brief summary of the required QC samples follows. The type and frequency of QC samples performed by the laboratory will be according to the specified analytical method.

6.4.1 Analytical Batch (Preparation Batch)

The analytical batch is defined as a set of samples that are extracted/analyzed concurrently or sequentially. The analytical batch will not exceed 20 samples. Significant gaps (greater than two hours) in the analytical sequence will result in the termination of the previous sequence and the initiation of a new analytical sequence. The analytical batch shall be analyzed sequentially on a single instrument. The practice of "holding a batch open" and performing a single set of batch QC samples for all analyses performed during that period is unacceptable.

The laboratory shall, at a minimum, analyze internal QC samples at the frequency specified in this QAPP for all analytical methods. These QC samples for each analytical batch shall include method blanks (MB) and laboratory control samples (LCS). Definitions for the QC samples described above are provided in Chapter 1, Update III to EPA SW-846. The matrix used for LCS analyses shall be reagent grade water for aqueous analyses and reagent sand for soil/sediment matrices.

Second column confirmation for all GC sample analyses involving identification of discrete peaks with detected concentrations will be required, as per the methods. Second column confirmation is not required for concentrations reported between the MDL and the QL.

6.4.2 Blanks

Two types of blanks routinely analyzed in the laboratory are method blanks and reagent blanks. Method blanks and reagent/solvent blanks are used to assess laboratory procedures as possible sources of sample contamination and can affect accuracy of the data.

Method or preparation blanks for all samples consist of deionized water or reagent sand that is subjected to the entire analytical procedure, including extraction, distillation, digestion, etc., as appropriate for the analytical method being utilized. One method blank will be analyzed for each analytical batch (minimum of one per day; one every 12 hours for GC/MS analyses). If the blank does not meet acceptance criteria, the source of contamination will be investigated and appropriate corrective action will be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on the sample results. Corrective actions may include reanalysis of the blank and/or repreparation and reanalysis of the blank and all associated samples. No method blank may exhibit a detected concentration greater than the quantitation limit. However, exceptions may be made when the analyte is not detected in the related sample. Sample results are not corrected for blank contamination unless required by the analytical method.

Reagent/solvent blanks consist of individual reagents or solvents subjected to the entire analytical procedure as appropriate for the analytical method being utilized. The blanks are only used if contamination problems are indicated by the method blank or if a new lot of materials are being checked before use.

6.4.3 Laboratory Control Samples

Laboratory control samples (LCS) are used as a means of evaluating the efficiency of the analytical process. As discussed above, LCS is used to generate precision and accuracy data that are compared, on a daily basis, to control limits. Laboratory control samples are subjected to the entire sample procedure, including extraction, digestion, etc., as appropriate for the analytical method utilized. They are generally introduced into an analytical batch (20 samples) immediately before extraction or analysis. LCS samples will be performed for both inorganic and organic laboratory methods.

6.4.4 Matrix Spikes and Matrix Spike Duplicates

A Matrix Spike (MS) is an environmental sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

A Matrix Spike Duplicate (MSD) is a duplicate of the environmental sample described above, each of which is spiked with known concentrations of analytes. The two spiked samples are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as relative percent difference (RPD) and percent recovery (%R).

6.4.5 Surrogate Recoveries and Standard Additions

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. Surrogates are added to samples to monitor the effect of the matrix on the accuracy of the analysis for each sample. Results are reported in percent recovery. Laboratories routinely add surrogates to samples requiring GC or GC/MS analysis and report these surrogate recoveries to the client. The laboratory does not modify its operations based on surrogate recoveries in environmental samples. However, obvious problems with sample preparation and analysis (e.g. evaporation to dryness, leaking septum, etc.) which can lead to poor surrogate spike recoveries must be ruled out prior to attributing low surrogate recoveries to matrix effects.

Standard Additions is the practice of adding a series of known amounts of an analyte to an environmental sample. The fortified samples are then analyzed and the recovery of the analytes calculated. The practice of standard addition is generally used with metals analysis and wet chemistry to determine the effect of the sample matrix on the accuracy of the analyses.

6.4.6 Calibration Standard

A calibration standard is prepared in the laboratory by dissolving a known amount of a purchased pure compound or standard mix in an appropriate matrix. The final concentration calculated from the known quantities is the true value of the standard. The results obtained from these standards are used to generate a standard curve and thereby quantify the compound in the environmental sample.

6.4.7 Reference Standard

A reference standard is prepared in the same manner as a calibration standard or may be obtained from National Institute of Standards and Testing (NIST). A reference standard is obtained from a source independent of the source of the calibration standard. The concentration of the known quantity is the "true" value of the standard. A reference standard is not carried through the same process used for the environmental samples, but is analyzed without digestion or extraction. A reference standard result is used to validate an existing concentration calibration standard file or calibration curve. The reference standard can provide information on the accuracy of the instrumental analytical method independent of various sample matrices.

6.5 Sensitivity

6.5.1 Method Detection Limit (MDL)

The method detection limit (MDL) is the lowest concentration at which a specific analyte in a matrix can be measured and reported with 99-percent confidence that the analyte concentration is greater than zero. MDLs are experimentally determined for each target analyte of the method. Each individual instrument will maintain a current MDL study. MDLs are based on the results of seven spikes of clean matrix at the estimated MDL and are statistically calculated in accordance with the Title 40, Code of Federal Regulations Part 136 (40 CFR 136), Attachment B. The standard deviation of the seven replicates is determined and multiplied by 3.143 (i.e., the 99-percent confidence interval from the one-sided student t-test). The MDLs are updated annually and whenever significant instrument maintenance is performed (i.e., GC Column, AA lamp, etc.).

6.5.2 Quantitation Limit (QL)

The quantitation limit is defined by the lowest concentration in the multi-point initial calibration. The QL is the lowest level for quantitation decisions based on individual measurements for a given method and representative matrix. The QL for this project is based on a project-specific action level and the capability of the method and laboratory. Detected results above the MDL but below the QL, are qualified with a J flag due to the very low comparator values. The J flag will denote the sample results as below the QL and as qualitative, estimated concentrations. This increases the probability of false positive results at these low concentrations, especially for the sample matrix anticipated for this project. However, analyst judgment will be used to determine if an apparent detected value should be reported or appears to be a false positive due to the sample matrix (e.g., from baseline "noise").

If dilution to bring the reported concentration of a single compound of interest within the linear range of the calibration, results in non-detect values for all other analytes with detected concentrations in the initial sample analysis, the results of the original run and the dilution will be reported with appropriate notations in the narrative of the report. Matrix effects (i.e., highly contaminated samples requiring dilution for analysis, dilution to bring detected levels within the range of calibration, and matrix interference requiring elevation of detection limits) will be considered in assessing compliance with the requirements for sensitivity. Cleanup procedures will be used to minimize interferences and lower the QLs to those required. In addition, the sample aliquot will be increased from the standard mass to make up for the increased QLs when data is reported on a dry weight basis (these samples are expected to be at least 50% moisture). This increased aliquot size may also increase the matrix interferences, as they too will have increased in mass. The QLs required by this project are listed in the method-specific tables in Attachment B of this document.

6.6 Corrective Action

The Sampling Team Leader is responsible for initiating corrective action and for implementation of all corrective actions with respect to the field sampling operations. The laboratory QA Director in consultation with the Project Chemist is responsible for implementing corrective actions in the laboratory. It is their combined responsibility to see that all analytical and sampling procedures are followed as specified and that the data generated meet the acceptance criteria. The acceptance criteria for some of the QC samples (LCS, surrogate recoveries) will be those calculated by the laboratory as control limits. The number of samples used to develop the statistical control limits shall be all those analyzed within the previous six months or a minimum of 20 datapoints. The comparison control limits in Attachment A are to ensure that the laboratory can produce data with acceptable accuracy. If the laboratory statistical limits are consistently different from the comparison limits, a different laboratory shall be selected for that analytical method, or an alternate analytical or preparation method shall be selected that increases the accuracy of the laboratory. Corrective action procedures are summarized for each method in Attachment A.

Corrective actions for the laboratory may include, but are not limited to:

- Reanalyzing samples;
- Correcting laboratory procedures;

- Recalibrating instruments using freshly prepared standards;
- Replacing solvents or other reagents that give unacceptable blank values;
- Training laboratory personnel in correct sample preparation and analysis procedures; and
- Accepting data with an acknowledged and documented level of uncertainty.

Whenever corrective action is deemed necessary, the Laboratory Director will ensure that the following steps are taken:

- The problem is defined;
- The cause of the problem is investigated and determined;
- Appropriate corrective action is determined; and
- Corrective action is implemented and its effectiveness verified.

6.7 Documentation

All calibration information, instrument maintenance and repair are recorded by the laboratory on appropriate forms developed for SW-846 procedures. Out-of-control analyses are generally described on a QA/QC discrepancy form and submitted to the laboratory supervisor for corrective action. Copies are distributed to the laboratory QA coordinator and laboratory director for approval, and to the case file. The calibration information is filed with the raw data.

7.0 DATA REDUCTION, VERIFICATION AND REPORTING

7.1 Laboratory Activities

7.1.1 Data Reduction and Verification

All analytical data generated within the laboratories shall be reviewed prior to report generation to assure the validity of the reported data. The data verification process consists of data generation, reduction, and three levels of documented review. In each stage, the review process will be documented by the signature of the reviewer and the date reviewed.

The analyst who generates the analytical data will have the prime responsibility for the correctness and completeness of the data. All data will be generated and reduced following protocols specified in laboratory SOPs. Each analyst will review the quality of his or her work based on an established set of guidelines outlined in the SOPs. The analyst will review the data package to ensure that:

- The correct samples were analyzed and reported in appropriate units,
- Preservation and holding time requirements were met,
- Sample preparation information is correct and complete,
- Appropriate SOPs have been followed,
- Analytical results are correct and complete,
- QC samples are within established control limits,
- Blanks are within appropriate QC limits,
- Special sample preparation and analytical requirements have been met, and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, anomaly forms are complete; holding times are documented, etc.).

The data reduction and validation steps shall be documented, signed and dated by the analyst. The analyst will then pass the data package to an independent reviewer, who will perform an independent review of the data package. This review is also to be conducted according to an established set of guidelines and to be structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented,

- QC samples are within established guidelines,
- Qualitative identification of sample components is correct
- Quantitative results are correct,
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; anomaly forms are complete; holding times are documented, etc.), and
- The data are ready for incorporation into the final report; and the data package is complete and ready for data archive.

The review is to be structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples will be checked to the bench sheet. This process will continue until no errors are found or until the data package has been reviewed in its entirety.

Data reviews shall be documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared. Before the report is released to the client, the data are reviewed for completeness and to ensure that the data satisfy the overall objectives of the project. The Laboratory Project Manager typically does this review.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

7.1.2 Data Reporting

At the conclusion of all analytical work for this project, the primary laboratory will submit a comprehensive certificate of analysis. The final certificates of analysis will be submitted no later than 21 days after the last sample has been submitted to the laboratory for the project. All samples shall be reported in a legally defensible package and electronic data deliverable (EDD) format consistent with the USACE, Sacramento District Automated Data Review (ADR) format. The data package may be submitted in a read-only electronic file, compatible with Adobe

Acrobat reader.

The data package for organics analyses will consist of a case narrative, chain-of-custody documentation, cooler receipt form, summary of results for environmental samples, summary of QA/QC results, and the data. Legible copies of all data will be organized systematically on numbered pages. The data for compound identification and quantitation must be sufficient to support all results presented in other sections of the data package. This section of the data package will include legible copies of the data for environmental samples (arranged in increasing order of field ID), and instrument calibration, QA/QC analyses, sample extraction and cleanup logs, instrument analysis logs for each instrument used. Instrument analysis logs are particularly important because they provide the basic link between all sample analyses and QC information (calibration, matrix spike, etc.). Instrument analysis logs for all instruments used for sample data for each analysis will include measurement printouts and quantitation reports for each instrument used.

Raw data will be available for further inspection, if required, and maintained in the central job file. All records related to the analytical effort are maintained at the primary laboratory in secured filing cabinets (i.e., cost information, scheduling, and custody). All records are maintained for five years after the final report is issued. Types of records to be maintained for the project include the following:

- Chain-of-custody records, including: information on the sampler's name, date of sampling, type of sampling, location of sampling, location of sampling station, number and type of containers used, signature of sampler relinquishing samples to non-contract personnel (e.g., Federal Express agent) with the date and time of transfer noted, signature of primary laboratory sample custodian receiving samples with date and time noted
- Cooler receipt form documenting sample conditions upon arrival at the laboratory.
- Any discrepancy/deficiency report forms due to problems encountered during sampling, transportation, or analysis
- Sample destruction authorization forms containing information on the manner of final disposal of samples upon completion of analysis
- All laboratory notebooks including raw data readings, calibration details, QC checks, etc

- Data system printouts (chromatograms, mass spectra, ICP data files, etc.)
- Tabulation of analytical results with supporting quality control information

7.1.2.1 Case Narrative

The case narrative will be written and the laboratory director or his/her designee will authorize the release of data. Items to be included in the case narrative are the field sample ID with the corresponding laboratory ID, parameters analyzed in each sample and the methodology used (EPA method numbers or other citation), detailed description of all problems encountered and corrective actions taken, discussion of possible reasons for out-of-control QA/QC results, and observations regarding any occurrences which may affect sample integrity or data quality.

7.1.2.2 Chain-of-Custody Documentation

Legible copies of chain-of-custody forms for each sample will be maintained in the data package. Cooler log-in sheets will be associated with the corresponding chain-of-custody form. Any integral laboratory-tracking document will also be included.

7.1.2.3 Summary of Environmental Results

For each environmental sample analysis, this summary shall include field ID and corresponding laboratory ID, sample matrix, date of sample extraction (if applicable), date and time of analysis, identification of the instrument used for analysis, instrument specifications, weight or volume of the sample used for analysis/extraction, dilution or concentration factor used for the sample extract, method detection limit or sample quantitation limit, definitions of any data qualifiers used, and analytical results.

7.1.2.4 Summary of QA/QC Results

The following QA/QC results will be presented in summary form. Details specified in Section 7.1.2.3 also will be included for the summary of QA/QC results. Acceptance limits for all categories of QC criteria will be provided with the data.

7.1.2.4.1 Organic Analyses (General)

The summary of QA/QC results for organic analyses will include:

- Initial Calibration - The concentrations of the standards used for analysis and the date and time of analysis. The response factor, percent relative standard deviation (%RSD), and retention time for each analyte (as applicable, GC, HPLC and GC/MS analyses) will be included in initial calibration summaries. A statement should also be made about the samples or dates for which a single initial calibration applies.

- Daily Calibration and Mid-level Standard - The concentration of the calibration standard used for daily calibration and/or the mid-level calibration check will be reported. The response factor, percent difference, and retention time for each analyte will be reported (GC and GC/MS). Daily calibration information will be linked to sample analyses by summary.
- Method Blank Analyses - The concentrations of any analytes found in method blanks will be reported even if detected amounts are less than the QL. The environmental samples and QA/QC analyses associated with each method blank will be stated.
- Surrogate Standard Recovery - The name and concentration of each surrogate compound added will be detailed. The percent recovery of each surrogate compound in the samples, method blanks, matrix spike/matrix spike duplicates and other QA/QC analyses will be summarized with sample IDs such that the information can be linked to sample and QA/QC analyses.
- Precision and Accuracy - For matrix spike/matrix spike duplicate analyses, the sample results, spiked sample results, percent recovery, and RPD with the associated control limits will be detailed. For laboratory duplicate analyses, the RPD between duplicate analyses will be reported as applicable. For laboratory QC check and/or LCS analyses, the percent recovery and acceptable control limits for each analyte will be reported. All batch QC information will be linked to the corresponding sample groups.
- Compound Identification (GC, HPLC, GC/MS): The retention times and the concentrations of each analyte detected in environmental and QC/QC samples will be reported for both primary and confirmation analyses. Mass spectra will also be included for reported detections in samples and for detections identified in the quantitation report, but ruled out during analyst review.
- Method Detection Limit (MDL): The MDL study result sheet will have laboratory heading, instrument identification, analysis date, spike level, average recovery, standard deviation and calculated MDL for each analyte.

In addition, the summary of QA/QC results for organic analyses will include the following information relating specifically to the method used.

7.1.2.4.2 GC and GC/MS Analyses

This section of the data package will include legible copies of the data for environmental samples (arranged in increasing order of field ID, primary and confirmation analyses). The raw data for each analysis will include chromatograms (with target compound, internal standard, and surrogate compounds labeled by name) with a quantitation report and/or area printout. GC/MS analyses will also include the mass spectra or ion chromatograms for each reported analyte.

7.1.2.4.3 Inorganic Analyses

The summary of QA/QC results for the inorganic analyses will include:

- Initial Calibration: The source of the calibration standards, true value concentrations, found concentrations, the percent recovery for each element analyzed, and the date and time of analysis will be reported.
- Continuing Calibration Verification: The source of the calibration standard, true value concentrations, found concentrations, the percent recovery for each element analyzed, and the date and time of analysis will be reported.
- Method Blank Analyses: The concentrations of any analytes found in initial calibration, continuing calibration blank, and in the preparation blank will be reported. The date and time of analysis also will be reported.
- Precision and Accuracy - Matrix Spikes and Sample Duplicates: For matrix spike analyses, the sample results, spiked sample results, percent recovery, spiking solution used, and the control range for each element will be detailed. For post digestion spikes, the concentrations of the spiked sample, the sample result, the spiking solution added, and recovery and control limits will be detailed. For laboratory duplicates, the original concentration, duplicate concentration, relative percent difference, and control limits will be detailed. Date and time for all analyses will be recorded.
- Precision and Accuracy - Laboratory Control Samples: The source of the laboratory control sample, true value concentrations, found concentrations, percent recovery for each element analyzed, and the date and time of analysis will be reported.

- Method of Standard Additions (MSA): This summary must be included when MSA analyses are required for analysis by Graphite Furnace AA. The absorbance values and the corresponding concentration values, the final analyte concentrations, and correlation coefficients will be reported for all analyses. Date and time of analysis will be recorded for all analyses.
- Method Detection Limit (MDL): The MDL study result sheet will have laboratory heading, instrument identification, analysis date, spike level, average recovery, standard deviation and calculated MDL for each analyte.

7.2 Field Activities

7.2.1 Data Reduction

Since no field screening equipment will be used during this sampling event, data reduction is not applicable.

7.2.2 Data Integrity

Integrity of information and data on field activities shall be maintained by the Project Leader. Integrity of the field sample custody is accomplished by the field staff, according to the sample custody procedures discussed in Section 5.0. This information is generated in the field and recorded in the project field logbook and on the sample chain-of-custody form, shall be verified before sample shipping, and confirmed at the laboratory upon their receipt of the samples.

7.2.3 Quality Assurance of Field Data

Validation of information and data on field activities shall be conducted as a QC procedure by the Technical Team Leader. The Technical Team Leader shall review laboratory results and field data before use. Chain-of-custody forms shall be cross-checked to the laboratory results to assure conformity of sample identification numbers. This information is compared to results of duplicate and blank samples, and field conditions at the time of sample collection will be taken into account when qualifying the sample analytical results, if applicable. Many of these cross-checks may be handled electronically.

7.2.4 Data Storage

Field and laboratory data shall be stored in hard copy and electronic format (when applicable) as part of the project file. This information is retained in the project file until project completion and closeout. Upon project closeout, all records shall be archived for permanent storage.

8.0 PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. Each laboratory shall have detailed SOPs on file that describe preventive maintenance procedures and schedules. All service and maintenance will be conducted by qualified laboratory staff or under service agreement with the manufacturer or their approved agent. All repairs, adjustments, and calibrations will be documented in a maintenance notebook or data sheet that will be maintained in a permanent file. The instrument notebook will clearly document the date, the problem description, corrective action taken, results of actions, and the name of the person performing the work. Table 8-1 lists common laboratory preventative maintenance parameters for laboratory instrumentation.

Table 8-1. Routine Laboratory Instrument Maintenance

Instrument	Operation	Frequency
Gas Chromatography	Change septum Change injection port liner Change column Bake detectors	Daily when used Daily when used As needed (when standard response decreases or sample carryover is noted, approximately monthly) As needed (when standard response decreases or sample carryover is noted , approximately monthly)
GC/MS	Clean source	As needed (show reduced sensitivity)
Atomic Absorption Spectrometer	Warm up instrument for 30 min. Digital readout values checked; check gas flows, cell alignment, wavelength, Photo multiplier voltage and lamp voltage Tygon tubing replaced Change contact rings Replace optical lens	Daily when used Daily when used Quarterly or as needed Daily, as needed or when used 6 months, or if deterioration is observed
Balances	Calibrate by manufacturer	Annually / verify monthly
Ovens/Refrigerators	Check temperature	Daily

9.0 ASSESSMENTS

9.1 Laboratory and Field Audits

All laboratories analyzing samples from the USACE are required to be USACE validated or to pass a National Environmental Laboratory Accreditation Committee (NELAC) audit. USACE validation is an evaluation of laboratory procedures or documentation and includes initial and periodic laboratory audits. The USACE laboratory on-site inspections or audits are performed by USACE chemists from the Center of Excellence in Omaha, Nebraska. The inspectors verify the following:

- The organization and personnel are qualified to perform assigned tasks,
- Adequate facilities and equipment are available,
- Complete documentation, included chain-of-custody of samples, is being implemented,
- Proper analytical methodology is being used without deviations, adequate analytical quality control (including reference samples, control charts, documented corrective actions, etc.) is being provided,
- Acceptable data handling and documentation techniques are being used,
- Adequate facilities and operations are installed to ensure laboratory health and safety, and
- Proper waste disposal procedures are implemented.

The on-site laboratory inspection helps to ensure that the laboratory is technically competent and that all the necessary quality control is being applied by the laboratory in order to deliver a quality product.

9.2 Laboratory Performance Evaluation Samples

At a minimum, the contract laboratory will participate in at least one performance evaluation program.

The performance evaluation (PE) samples are single blind (prepared by the laboratory from ambulated standards) and are often associated with the regular laboratory audits performed by the USACE and/or regulatory agencies. USACE, Center of Excellence, Omaha, Nebraska reviews the results of the PE samples to determine if the laboratory should continue to receive USACE validation.

9.3 Quality Assurance Samples

QA samples are replicate samples submitted to a different laboratory, and subjected to the same environmental conditions and steps in the measurement process as the primary sample. They serve as an oversight function in assessing the analytical portion of the measurements system. QA samples will be collected once during the SI field effort for the groundwater samples.

9.4 Data Validation

The laboratory data will be validated using guidelines in Appendix C. The validation guidelines are based on EPA SW-846 methods and the EPA National Functional Guidelines for Organic and Inorganic Data Review. The Appendix C procedures shall supercede the procedures in these references. However, professional judgment shall be used when deciding if qualification of data is applicable. When professional judgment is applied that differs from the qualification scheme in Appendix C, the rationale shall be provided in the validation report. The data will be validated using an automated data review (ADR) software package prepared for the USACE. A chemist will review the data to ensure that the proper qualifiers have been added to the data.

9.5 Data Quality and Usability Assessment

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its PARCC parameters. These terms are described as follows:

Precision

Precision is a measure of the reproducibility of analyses under a given set of conditions. Precision will be assessed by the RPD from replicate measurements of duplicate control samples, reference materials, or environmental samples.

Accuracy

Accuracy is a determination of how close the measurement is to the true value. Accuracy will be assessed by the percent recovery of spiked blank or environmental samples and by any external contamination evident from any associated field or laboratory blank results.

Representativeness

Representativeness is a qualitative parameter that reflects the extent to which a given sample is characteristic of a given population at a specific location or under a given environmental condition. Representativeness is best satisfied by making certain that sampling locations are selected properly, a sufficient number of samples are collected, and an appropriate sampling technique is employed. Variations at a sampling point will be evaluated based on the results of field duplicates. Some samples may require analysis of multiple phases to obtain representative results. Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. Sample representativeness will also be evaluated based on results from method blanks and trip blanks.

Completeness

Completeness will be evaluated qualitatively and quantitatively. The qualitative evaluation of completeness will be determined as a function of all events contributing to the sampling event including items such as correct handling of COC forms, incorporation of QC samples at the appropriate frequency, etc. The quantitative description of completeness is defined as the percentage of acceptable QC parameters that can be controlled. The goals for completeness are as follows: contract (95%), analytical (85%), technical (95%), and field sampling completeness (100%). Contract completeness is a measure of the results that meets contract requirements relative to the number of reported results expressed as a percentage. Analytical completeness is a measure of all unqualified results relative to the number of reported results expressed as a percentage. Qualified data due to detections below the QL will NOT count against analytical completeness. Technical completeness is a measure of the usable results relative to the number of reported results expressed as a percentage. Field sampling completeness is a measure of the number of samples collected relative to the number of samples planned expressed as a percentage.

Comparability

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. To ensure comparability, field procedures will be standardized and field operations will adhere to standard operating procedures. Laboratory data comparability will be assured by use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), and consistency in reporting units (μg/L, mg/Kg, etc.). Analysis of standard reference materials will follow EPA or other standard analytical methods, which utilize standard units of measurement, methods of analysis, and reporting format.

The Project Chemist will discuss the PARCC parameters of the project data and the impact on data usability in a Chemical Data Quality Assessment Report (CDQAR). The CDQAR will include an introduction, field procedures, number of samples collected and associated parameters, analytical methods used, frequency and compliance with criteria in this QAPP, impact of any non-compliant data, any systematic problems with the dataset, discussion of field duplicate results and impact on variability of the chemical concentration, and a brief conclusion regarding the usability of the data. This usability will relate directly to the objectives addressed in the Data Quality Objectives. The CDQAR will be presented as an appendix to the report.

10.0 REFERENCES

10.1 Environmental Protection Agency (EPA)

EPA 2001. *EPA Requirements for Quality Assurance Project Plans,* EPA QA/R-5, Final Interim Final, March.

EPA 2000a. *Guidance for Data Assessment*, USEPA QA/G-9, Final, July.

EPA 2000b. *Guidance on the Data Quality Objectives Process*, USEPA QA/G-4, Final, September.

EPA 1998. *Test Methods for Evaluating Solid Waste*, USEPA SW-846, Third Edition, (Update III), June.

National Functional Guidelines for Inorganics Data Review, USEPA Contract Laboratory Program, EPA 540/R-94/013.

National Functional Guidelines for Organic Data Review, USEPA Contract Laboratory Program, EPA 540/R-94/012.

10.2 U.S. Army Corps of Engineers (USACE)

Requirements for the Preparation of Sampling and Analysis Plans, Engineering Manual EM. 200-1-3, 1998.

Chemical Data Quality Management for Hazardous Waste Remedial Activities, Engineering Regulation 1110-1-263, October 1990.

ATTACHMENT A

CALIBRATION AND QUALITY CONTROL PROCEDURES

ACRONYMS AND ABBREVIATIONS

CCC	Calibration check compound
CCV	Continuing calibration verification standard
COD	Coefficient of determination
CV	Calibration verification standard
%D	Percent difference
GC	Gas chromatography
GC/MS	Gas chromatography/mass spectrometry
ICS	Interference check standard
ICV	Initial calibration verification standard
MDL	Method detection limit
MS/MSD	Matrix spike/matrix spike duplicate
LCS	Laboratory control sample
QC	Quality control
QL	Quantitation limit
r	Correlation coefficient
r^2	Coefficient of determination
RF	Response factor
RPD	Relative percent difference
RRF	Relative response factor
RSD	Relative standard deviation
SIM	Selective Ion Monitoring
SPCC	System performance check compound
USACE	United States Army Corps of Engineers

Table A-1
Summary of Calibration and Internal Quality Control Procedures for Method SW8081A (Total DDTs)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW8081A	DDD, DDE, DDT	Five-point initial calibration	Prior to sample analysis and when CCV fails	Option 1: RSD for each analyte ≤ 20% Option 2: Grand mean RSD ≤ 20%, with no individual analyte RSD >30% Option 3: Linear regression – r ≥ 0.995 Option 4: Non-linear regression COD $r^2 \geq 0.990$ (6 points for 2nd order, 7 points for 3rd order)	Correct problem then repeat initial calibration.
		Second source standard (not required if calibration verification below is prepared with a second source of the standard)	Following initial calibration	% Difference from expected value ≤ 15% for all analytes OR grand mean ≤ 15% with no individual response factor greater than 20%	Correct problem, rerun second source standard. If that fails, repeat initial calibration.
		DDT and endrin breakdown check	Daily prior to analysis of samples	Degradation < 15%	Correct problem, then repeat breakdown check.
		Calibration verification	ICV: At the beginning of an analysis sequence CCV: After every 10 field samples and at the end of the analysis sequence	Response factor for all analytes within ±15% of initial calibration response factor OR grand mean within 15% with no individual response factor greater than 25%	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration CCV: Correct problem, then repeat CCV and reanalyze all samples since last successful CCV or ICV

Table A-1
Summary of Calibration and Internal Quality Control Procedures for Method SW8081A (Total DDTs)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW8081A	DDD, DDE, and DDT	Method Blank	1 per preparation batch	All analytes < ½ QL.	Investigate possible contamination source. Take appropriate corrective action. Reprepare and reanalyze all samples processed with a contaminated blank, unless analyte is not detected in associated samples or present at greater than 10x blank concentration.
		Laboratory Control Sample	1 per preparation batch	Comparison recovery limits 50-130% (soil)	Correct problem, then reprepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes.
		Matrix Spike and Matrix Spike Duplicate	1 MS/MSD per 20 project samples when identified on the Chain-of-Custody	Comparison recovery limits 50-130% (soil) and RPD <35% for soil samples	Evaluate for supportable matrix effect. If no interference is evident reprepare and reanalyze MS/MSD and all samples in the preparation batch once within the holding time. If still out report both sets of data.
		Surrogate spike	All field and quality control samples	Comparison recovery limits 50-130% (soil)	Evaluate for supportable matrix effect. If no interference is evident reprepare and reanalyze affected sample(s).
		Confirmation of positive results (second column or second detector)	All detected results at or above the QL must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and secondary column RPD $\leq$ 40%	None – report as detected result if criteria is met. Use professional judgment to determine whether primary or secondary column concentration should be reported. Report as not detected at QL if criteria is not met.
		Quantitation limit standard (lowest concentration on initial calibration curve)	Verify at least once for every matrix and field effort	QLs established shall not exceed those in the Appendix B tables.	QLs that exceed established criteria shall be submitted to USACE Project Chemist for approval prior to analysis of any project samples.

Table A-2
Summary of Calibration and Internal Quality Control Procedures for Modified Method SW8270 (Polynuclear Aromatic Hydrocarbons by GC/MS Selective Ion Monitoring)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW8270 SIM	Polynuclear Aromatic Hydrocarbons	Instrument tune (decafluorotriphenyl-phosphine)	Prior to initial calibration and every 12 hours of analysis time	Ion abundance criteria as described in SW8270C	Retune instrument and verify. Rerun affected samples.
		Five-point calibration	When daily calibration verification fails or following major instrument maintenance or repair	1. Average RRF for SPCCs: ≥ 0.050. 2. %RSD for RRFs for CCCs: ≤30% 3. One option below for ALL analytes; Option 1: RSD for each analyte ≤15% Option 2: Grand mean ≤15% with no individual analyte RSD >30% Option 3: linear regression, r≥ 0.995 Option 4: non-linear regression – COD $r^2 \geq 0.990$ (6 points 2nd order, 7 points 3rd order)	Correct problem then repeat initial calibration.
		Second source calibration verification	Once after each initial calibration	% Difference from expected value ≤ 25% for all analytes.	Correct problem and verify second source standard. If that fails, then repeat initial calibration.

Table A-2
Summary of Calibration and Internal Quality Control Procedures for Modified Method SW8270 (Polynuclear Aromatic Hydrocarbons by GC/MS Selective Ion Monitoring)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW8270 SIM	Polynuclear Aromatic Hydrocarbons	Calibration verification	Daily, prior to sample analysis and every 12 hours of analysis time	1. Average RRF for SPCCs: ≥ 0.050 2. %Difference/drift for CCCs: ≤ 20%D 3. Grand mean of concentration for all analytes within ±20%D of expected value, with no individual analytes (except CCCs) > 25%.	Correct problem, rerun CV. If that fails, then repeat initial calibration.
		Calibration verification internal standards	With every CV	Retention time ± 30 seconds from retention time of midpoint standard in the initial calibration. Quantitation ion peak area within 2 times area of initial calibration midpoint standard	Inspect mass spectrometer and GC for malfunctions. Take appropriate corrective actions. Reanalyze samples analyzed while system was malfunctioning.
		Method Blank	1 per preparation batch	All analytes < ½ QL. For common laboratory contaminants, all analytes < QL.	Investigate possible contamination source. Take appropriate corrective action. Reprepare and reanalyze all samples processed with a contaminated blank, unless analyte is not detected in associated samples or present at greater than 10x blank concentration.
		Laboratory Control Sample	1 per preparation batch	Comparison recovery limits - 50-120%	Correct problem, then reprepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes.

Table A-2
Summary of Calibration and Internal Quality Control Procedures for Modified Method SW8270 (Polynuclear Aromatic Hydrocarbons by GC/MS Selective Ion Monitoring)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW8270 SIM	Polynuclear Aromatic Hydrocarbons	Matrix Spike and Matrix Spike Duplicate	1 MS/MSD per 20 project samples when identified on the Chain-of-Custody	Comparison recovery limits 50-120% and RPD <35 %	Evaluate for supportable matrix effect. If no interference is evident reprepare and reanalyze MS/MSD and all affected samples once within the holding time. If still out report both sets of data.
		Surrogate spike	All field and quality control samples	Comparison recovery limits 50-120%	Evaluate for supportable matrix effect. If no interference is evident reprepare and reanalyze affected sample(s).
		Quantitation limit standard (lowest concentration on initial calibration curve)	Verify at least once for every matrix and field effort	QLs established shall not exceed those in the Appendix B tables.	QLs that exceed established criteria shall be submitted to USACE Project Chemist for approval prior to analysis of any project samples.

Table A-3
Summary of Calibration and Internal Quality Control Procedures for Method SW6010B (Selected Metals)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW6010B	Selected Metals	Initial calibration (minimum one high standard and a blank)	Daily prior to sample analysis	None unless run more standards; then r > 0.995	Correct problem and repeat initial calibration.
		Initial calibration verification standard (equivalent to second source standard)	Once after initial calibration, prior to sample analysis	% Difference from expected value ≤ 10% for all analytes	Correct problem, rerun ICV (and all samples run since the ICV, if applicable). If that fails, repeat initial calibration.
		Continuing calibration verification	After every 10 field samples and at the end of the analysis sequence	% Difference from expected value ≤ 10% for all analytes	Correct problem, then repeat CCV and reanalyze all samples since last successful CCV or ICV. If that fails repeat initial calibration.
		Low-level calibration check standard	At the beginning of the analysis sequence if one standard initial calibration used.	% Difference from expected value ≤ 30% for all analytes	Correct problem, then reanalyze.
		Calibration blanks	Initial calibration blank (ICB): At the beginning of an analysis sequence following the ICV. Continuing calibration blank (CCB): After every 10 field samples and at end of sequence following the CCV.	All analytes ≤ ½ QL	ICB: Correct problem, rerun ICB (and all samples run since the ICB, if applicable). CCB: Correct problem, then repeat CCB and reanalyze all samples since last successful CCB or ICB.
		Method Blank	1 per preparation batch	All analytes ≤ ½ QL	Correct problem, then reprepare and reanalyze all samples in the preparation batch unless analyte is not detected in associated samples or present at greater than 10x blank concentration.

Table A-3
Summary of Calibration and Internal Quality Control Procedures for Method SW6010B (Selected Metals)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW6010B	Selected Metals	Interference check standard solutions (ICS)	At the beginning of the analytical sequence	% Difference from expected value ≤ 20% for all analytes	Correct problem, then reanalyze ICS and associated samples.
		Laboratory control sample	1 per preparation batch	Comparison recovery limits 80-120%	Correct problem, then reprepare and reanalyze LCS and all samples in the associated preparatory batch for failed analytes.
		Matrix Spike (level of spike must be less than the mid-level standard of the calibration curve)	1 MS per 20 project samples when identified on the Chain-of-Custody	Comparison recovery limits 80-120%	Evaluate for supportable matrix effect. If no interference is evident reprepare and reanalyze MS/MSD and all affected samples once within the holding time. If still out report both sets of data.
		Matrix Duplicate or Matrix Spike Duplicate	1 duplicate per preparation batch or 1 MSD per 20 project samples when identified on the Chain-of-Custody	RPD <25	Evaluate for supportable matrix effect. If no interference is evident reprepare and reanalyze MS/MSD and all affected samples once within the holding time. If still out report both sets of data.
		Serial Dilution (1:5 dilution)	Each preparation batch or when a new matrix is encountered and result is > 25 times the method detection limit (MDL)	Agreement between undiluted and diluted results ±10%	Perform post digestion spike
		Post digestion spike	When serial dilution fails	All analytes recovered 75-125% of expected result	Perform method of standard addition for all samples with similar matrix
		Method of Standard Addition	As needed for samples with confirmed matrix effects	r ≥ 0.995	Consider alternative sample preparation or analysis methods to reduce interference and discuss with USACE Project Chemist
		Quantitation limit standard (lowest concentration on initial calibration curve)	Verify at least once for every matrix and field effort	QLs established shall not exceed those in the Appendix B tables.	QLs that exceed established criteria shall be submitted to USACE Project Chemist for approval prior to analysis of any project samples.

Table A-4
Summary of Calibration and Internal Quality Control Procedures for Method SW7471A (Mercury)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW7471A	Mercury	Calibration (5 standards and blank)	Daily	r > 0.995	1) Identify and repeat analysis for outlying points 2) Recalculate using valid points
		ICV/CCV	Daily: before sample analysis, every 10 samples, and at the end of the analytical sequence	ICV: % Recovery $\pm$10% CCV: % Recovery $\pm$20%	1) Reanalyze ICV/CCV 2) If still out, identify and correct problem 3) Recalibrate and reanalyze all samples since last valid CCV
		ICB/CCB	Beginning of sequence, every 10 samples, and at end of sequence	Analytes < MDL	1) Reanalyze ICB/CCB 2) If still out, identify and correct problem 3) Recalibrate and reanalyze all samples since last valid CCB
		Method Blank (MB)	1 per sample preparation batch	Analytes < ½ QL	1) Investigate possible contamination source 2) Take appropriate corrective action 3) Repeat instrument blank analysis 4) Redigest and reanalyze all samples processed with a contaminated blank at no cost to USACE, unless analyte is not detected in associated samples or present at greater than 10x blank concentration. 5) Flag sample results associated with blank contamination
		LCS	1 per sample preparation batch	Comparison recovery limits 80-120%	1) Reanalyze LCS. 2) If still out identify and correct problem. 3) Reprepare and reanalyze affected samples.
		Matrix Spike (MS) (level of spike must be less than the mid-level standard of the calibration curve)	1 per preparation batch	Comparison recovery limits 80-120%	1) Evaluate for supportable matrix effect. 2) If no interference is evident re-extract and reanalyze MS once. 3) If still out report both sets of data.

Table A-4
Summary of Calibration and Internal Quality Control Procedures for Method SW7471A (Mercury)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW7471A	Mercury	Matrix Duplicate (D) or Matrix Spike Duplicate (MSD)	1 per sample batch	RPD <20	1) Recalculate result; if still out: 2) Evaluate for supportable matrix effect. 3) If no interference is evident reanalyze affected sample(s) and narrate any outliers.
		QL	Low point on initial calibration curve.	QLs established shall not exceed those required by project; Refer to accompanying table.	QLs that exceed established criteria shall be submitted to USACE for approval prior to any project samples analyses

ATTACHMENT B

MAXIMUM QUANTITATION LIMITS

Table B-1
Maximum Quantitation Limits (QLs) and Action Goals for Metals by Method SW6010B and Mercury by Method 7471A

Analyte	ROD/RAP Action Goals (mg/kg)	Soil QL (mg/Kg)	Soil MDL (mg/kg)
Antimony	None Established	10	1.7
Arsenic	16.7	5	0.4
Cadmium	1.2	0.50	0.1
Chromium	112	10	0.22
Copper	68.1	10	0.1
Lead	46.7	20	0.24
Mercury	0.43	0.1	0.033
Nickel	114	10	0.27
Zinc	158	10	0.19

Table B-2
Maximum Quantitation Limits (QLs) and Action Goals for Total DDTs by Method SW8081A

Analyte	Action Goal (mg/kg)	Soil QL (mg/kg)	Soil MDLs (mg/kg)
4,4'-DDD	0.024 (Total DDTs-revised)	0.005	0.001
4,4'-DDE	0.024 (Total DDTs- revised)	0.005	0.001
4,4'-DDT	0.024 (Total DDTs- revised)	0.005	0.001

These detection limits were calculated using a clean matrix and may not be achievable with the samples collected for this project. By reporting down to the detection limit, there is an increased probability of low level false positives.

Total DDTs = DDT + DDD + DDE

Table B-3
Maximum Quantitation Limits (QL) and Action Goals for Polynuclear Aromatic Hydrocarbons (PAHs) by Method SW8270, Selective Ion Monitoring (SIM)

Analyte	ROD/RAP Action Goals (mg/kg)	Soil QL (mg/kg)	Soil MDLs (mg/kg)
Acenaphthene	Not applicable	0.01	0.0015
Acenaphthylene	Not applicable	0.03	0.0015
Anthracene	Not applicable	0.04	0.0015
Benzo(a)anthracene	Not applicable	0.03	0.0015
Benzo(b)fluoranthene	Not applicable	0.03	0.0015
Benzo(k)fluoranthene	Not applicable	0.03	0.0015
Benzo(g,h,i)perylene	Not applicable	0.03	0.0015
Benzo(a)pyrene	Not applicable	0.03	0.0015
Chrysene	Not applicable	0.03	0.0015
Dibenz(a,h)anthracene	Not applicable	0.03	0.0015
Fluoranthene	Not applicable	0.03	0.0015
Fluorene	Not applicable	0.01	0.0015
Indeno(1,2,3-cd)pyrene	Not applicable	0.03	0.0015
2-Methylnaphthalene	Not applicable	0.01	0.0015
Naphthalene	Not applicable	0.03	0.0015
Phenanthrene	Not applicable	0.03	0.0015
Pyrene	Not applicable	0.03	0.0015
Sum of PAHs	4.022	Sum of QLs = 0.46	Not applicable

These detection limits were calculated using a clean matrix and may not be achievable with the samples collected for this project. By reporting down to the detection limit, there is an increased probability of low level false positives.

ATTACHMENT C

VALIDATION QUALIFIER CONVENTIONS

Table C-1
Default Data Qualifier Convention for GC Analyses

Quality Control Item	Evaluation	Data Qualifier Flag		Sample(s) Qualified
		Detects	Nondetects	
Holding Times (Extraction/Analysis)	1) Holding time exceeded by 2 times or less 2) Holding time exceeded by greater than 2 times	J- J-	UJ R	Sample
Cooler Temperature	1) > 6 and ≤10 degrees Centigrade 2) >10 degrees Centigrade 3) < 2 degrees Centigrade	J- J- No qual.	UJ R No qual.	All samples shipped in the affected cooler
Initial Calibration	1) %RSD > 20% 2) r < 0.995, r^2 <0.990	J J	UJ UJ	All samples run on the same instrument under that initial calibration
Initial and Continuing Calibration Verification (ICV and CCV) and Second Source Standard	1) % Difference > +20% 2) % Difference < -20% and ≥ -50% 3) % Difference < -50%	J+ J- J-	No qual. UJ R	All samples bracketed by the ICV, CCV or under initial calibration associated with second source standard
Method Blank Contamination	1) Sample results for common lab contaminant less than or equal to 10 times the blank contamination 2) Sample results for other compounds less than or equal to 5 times the blank contamination	U U	No qual. No qual.	All samples in the same preparation batch
Surrogate Recovery	1) % Recovery < control limit (CL) but ≥ 10% 2) % Recovery <10% 3) % Recovery > CL	J- J- J+	UJ R No qual.	Sample
Matrix Spikes	1) % Recovery < CL but ≥ 10% 2) % Recovery <10% 3) % Recovery > CL 4) RPD > CL	J- J- J+ J	UJ R No qual. UJ	Parent Sample
Laboratory Control Sample Recovery	1) % Recovery < CL but ≥ 10% 2) % Recovery <10% 3) % Recovery > CL 4) RPD > CL	J- J- J+ J	UJ R No qual. UJ	All samples in the same preparation batch
Quantitation Limits	Quantitation limits not matching the project specified limits. Results reported below the quantitation limit.	No qual. J	No qual. No qual.	Sample (note in validation report) Sample
Field Duplicates	RPD > 50 (soil)	No qual.	No qual.	Parent sample-review dataset for systematic occurrences

Table C-1
Default Data Qualifier Convention for GC Analyses

Quality Control Item	Evaluation	Data Qualifier Flag		Sample(s) Qualified
		Detects	Nondetects	
Equipment Blanks	1) Sample results for common lab contaminant less than or equal to 10 times the blank contamination	U	No qual.	All samples in the same sampling event
	2) Sample results for other compounds less than or equal to 5 times the blank contamination	U	No qual.	

Alternate qualifiers are acceptable on a case-by-case basis based upon validator professional judgment. All deviations from the above qualification scheme shall be documented in the validation report.

Control limits are specified in Appendix A.

Table C-2
Default Data Qualifier Convention for GC/MS Analyses

Quality Control Item	Evaluation	Data Qualifier Flag		Sample(s) Qualified
		Detects	Nondetects	
Holding Times (Extraction/Analysis)	1) Holding time exceeded by 2 times or less 2) Holding time exceeded by greater than 2 times	J- J-	UJ R	Sample
Cooler Temperature	1) > 6°C and ≤10°C 2) >10°C 3) < 2°C	J- J- No qual.	UJ R No qual.	All samples shipped in the affected cooler
Instrument Tuning	1) Ion abundance criteria not met	JN	R	All samples associated with an initial calibration, if tune is associated to an initial calibration. All samples in same instrument batch, if tune is associated with calibration verification.
Initial Calibration	1) Average RRF < criteria in Appendix A tables 2) %RSD > 30% 3) r < 0.995	J J J	R UJ UJ	All samples associated with the initial calibration
Second Source Standard and Continuing Calibration Verification (CCV)	1) Average RRF < criteria in Appendix A tables 2) % Difference > +25% 3) % Difference < -25% and ≥ -50% 4) % Difference < -50%	J J+ J- J-	R No qual. UJ R	All samples associated with the second source standard and CCV
Method Blank Contamination	1) Sample results for common lab contaminant less than or equal to 10 times the blank contamination 2) Sample results for other compounds less than or equal to 5 times the blank contamination	U U	No qual. No qual.	All samples in the same preparation batch
Surrogate Recovery	1) % Recovery < CL but ≥ 10% 2) % Recovery <10% 3) % Recovery > CL Note: For semivolatile analysis, two or more surrogates in a fraction must be out of criteria for qualification unless recovery < 10%.	J- J- J+	UJ R No qual.	Sample
Matrix Spike Recovery	1) % Recovery < CL but ≥ 10% 2) % Recovery <10% 3) % Recovery > CL 4) RPD > CL	J- J- J+ J	UJ R No qual. UJ	Parent Sample

Table C-2
Default Data Qualifier Convention for GC/MS Analyses

Quality Control Item	Evaluation	Data Qualifier Flag		Sample(s) Qualified
		Detects	Nondetects	
Laboratory Control Sample Recovery	1) % Recovery < CL but ≥ 10% 2) % Recovery <10% 3) % Recovery > CL 4) RPD > CL	J- J- J+ J	UJ R No qual. UJ	All samples in the same preparation batch
Quantitation Limits	Quantitation limits not matching the project specified limits. Results reported below the quantitation limit.	No qual. J	No qual. No qual.	Sample (note in validation report) Sample
Field Duplicates	RPD >50 (soil)	No qual.	No qual.	Parent sample-review dataset for systematic occurrences
Equipment Blanks	1) Sample results for common lab contaminant less than or equal to 10 times the blank contamination 2) Sample results for other compounds less than or equal to 5 times the blank contamination	U U	No qual. No qual.	All samples in the same sampling event

Alternate qualifiers are acceptable on a case-by-case basis based upon validator professional judgment. All deviations from the above qualification scheme shall be documented in the validation report.

Control limits are specified in Appendix A.

Table C-3
Default Data Qualifier Convention for Metals Analyses

Quality Control Item	Evaluation	Data Qualifier Flag		Sample(s) Qualified
		Detects	Nondetects	
Holding Times	1) Holding time exceeded by 2 times or less 2) Holding time exceeded by greater than 2 times	J- J-	UJ R	Sample
Initial Calibration	1) r < 0.995	J	UJ	All samples run on same instrument under the same calibration
Initial and Continuing Calibration Verification (ICV and CCV)	1) % Recovery > 110% but ≤ 125% 2) % Recovery > 125% 3) % Recovery < 90% but ≥75% 4) % Recovery < 75%	J+ R J- J-	No qual. No qual. UJ R	All samples bracketed by ICV or CCV
Method Blank Contamination	Sample results less than or equal to 5 times the blank contamination	U	No qual.	All samples in the same preparation batch
Matrix Spike Recovery	1) % Recovery < 80% but ≥ 30% 2) % Recovery <30% 3) % Recovery > 120% 4) RPD > 20	J- J- J+ J	UJ R No qual. UJ	All samples from same site and similar matrix interference
Laboratory Control Sample Recovery	1) % Recovery < 80% but ≥ 50% 2) % Recovery <50% 3) % Recovery > 120% 4) RPD > 20	J- J J+ J	UJ R No qual. UJ	All samples in the same preparation batch
Quantitation Limits	Quantitation limits not matching the project specified limits Reported result less than the quantitation limit.	No qual. J	No qual. No qual.	Sample (note in validation report) Sample
Field Duplicates	RPD >50 (soil)	No qual.	No qual.	Parent sample-review dataset for systematic occurrences
Equipment Blanks	Sample results within 5 times blank contamination	U	No qual.	All samples in the same sampling event

Alternate qualifiers are acceptable on a case-by-case basis based upon validator professional judgment. All deviations from the above qualification scheme shall be documented in the validation report.

ATTACHMENT D

IMMUNOASSAY TEST INSTRUCTIONS

STRATEGIC DIAGNOSTICS INC.
EnviroGard® DDT in Soil Test Kit
73100

Intended Use
The EnviroGard DDT in Soil Test Kit is a qualitative or semi-quantitative field test for the detection of DDT and its metabolites DDD and DDE in soil. The EnviroGard DDT in Soil Test Kit allows rapid semi-quantitative screening for DDT at 0.2, 1.0, and 10.0 parts per million (ppm) in soils.

Test Principles
The EnviroGard DDT in Soil Test Kit is based on the use of polyclonal antibodies that bind either DDT or DDT-Enzyme Conjugate. These antibodies are immobilized to the walls of the test tubes. When DDT is present in the sample, it competes with the DDT-Enzyme Conjugate for a limited number of antibody binding sites.

Since there are the same number of antibody binding sites on every test tube and each test tube receives the same number of DDT-Enzyme Conjugate molecules, a sample that contains a low concentration of DDT allows the antibody to bind many DDT-Enzyme Conjugate molecules.

Therefore, a low concentration of DDT produces a dark blue solution. Conversely, a high concentration of DDT allows fewer DDT-Enzyme Conjugate molecules to be bound by the antibodies, resulting in a lighter blue solution.

NOTE: Color is inversely proportional to DDT concentration.

Darker color = Lower concentration
Lighter color = Higher concentration

Performance Characteristics
The EnviroGard DDT in Soil Test Kit will not differentiate between DDT, its metabolites, and other structurally similar compounds, but will detect their presence to differing degrees. The following table shows a number of compounds and the approximate concentration of each required to yield a positive result (Lower Limit of Detection or LLD), and the concentration required to inhibit one-half of the color developed by the Negative Control (IC50). Concentration is in parts per million (ppm) in soil.

Compound	LLD	IC50
p,p'-DDT (kit calibrator)	0.04	1.25
p,p'-DDD	0.01	0.3
p,p'-DDE	0.18	3.6
o,p'-DDT	4	93
o,p'-DDD	0.4	11
o,p'-DDE	3	93
DDA	0.002	0.04
Chloropropylate	0.007	0.08
Chlorobenzilate	0.03	0.35
Dicofol	0.14	2
Tetradifon	1.2	14
Thiobencarb	5	52
Tebuconazole	7	95
Neburon	17	284
Chloroxuron	24	216
Monolinuron	25	714
Diclofop	70	>1000

The following compounds have lower limits of detection > 100 ppm:

2,4-D	4-chlorophenoxyacetic acid
Chlorbromuron	Chlordane
Chlortoluron	Dicamba
Diflubenzuron	Diuron
Lindane	Linuron
MCPA acid	MCPB
Mecoprop	

Precautions
• Treat DDT, solutions that contain DDT and potentially contaminated soil samples as hazardous materials.
• Where appropriate, use gloves, proper protective clothing, and methods to contain and handle hazardous material.
• Store all test kit components at 4°C to 8°C (39°F to 46°F) when not in use.
• Do not freeze test kit components or expose them to temperatures greater than 37°C (99°F).
• Allow all reagents to reach ambient temperature (18°C to 27°C or 64°F to 81°F) before beginning the test.
• Do not use test kit components after the expiration date.
• Do not use reagents or test tubes from one test kit with reagents or test tubes from a different test kit.
• Use approved methodologies to confirm any positive results.
• Do not dilute or adulterate test reagents or use samples not called for in the test procedure; this may give inaccurate results.
• Tightly recap the DDT calibrator vials to prevent evaporative loss.
• Distribution of DDT in soils may be highly variable. The use of a composite sampling technique may be appropriate. Development of a sampling plan that assures adequate sample number and distribution is the responsibility of the analyst.
• DDT is light sensitive. Store soil extracts at 2°C to 7°C, shielded from direct light.

Materials Provided
EnviroGard DDT in Soil Test Kit
This test kit contains the following items:
20 Antibody-Coated Test Tubes
1 vial of Assay Diluent
1 vial of Negative Control (methanol)
1 vial of 0.2 ppm DDT Calibrator in methanol
1 vial of 1.0 ppm DDT Calibrator in methanol
1 vial of 10.0 ppm DDT Calibrator in methanol
1 vial of DDT-Enzyme Conjugate
1 vial of Substrate
1 vial of Stop Solution
1 20-place Test Tube Rack

22 Pipette Tips, yellow (for the Gilson M-25 Microman® Positive Displacement Pipettor)

Materials Required but Not Provided
You will also need several other items, some of which are included in the EnviroGard Soil Field Lab.

- Methanol-ACS reagent grade Methanol is required for soil extraction, but is not included in the EnviroGard Soil Extraction Kit. You must order it separately.
- EnviroGard Soil Extraction Bottle Kit

Use this kit for the extraction of DDT in soil samples. This kit contains enough devices to process 14 samples:
• 14, 30 mL LDPE Bottles with screw caps (each bottle contains stainless steel mixing beads)
• 14 filtration caps
• 14 Millex® HV_{13} filters
• 18 Wooden Spatulas
• 1 Syringe with coupler
• 1 Syringe coupler
• 14 Screw Top Glass Vials, 4.0 mL
• 14 Stoppers

• 18 Weigh Boats
• Gilson M-25 Microman Positive Displacement Pipettor
• Eppendorf™ Repeater® Pipettor and five Combitips® (3 x 12.5 mL, 1 x 5.0 mL, and 1 x 50 mL)
• Balance capable of accurately weighing 5 grams
• Differential Photometer or RPA-I Photometer
• Indelible marker for labeling test tubes
• Watch or timer
• Clean running water or a wash bottle containing tap or deionized water (500 mL)
• Calculator (optional)

Suggestions for Pipettor Use

• Practice using both pipettors (positive displacement and Repeater pipettor) with water and extra tips before you analyze your samples.
• Use a new tip each time you use the Repeater pipettor to avoid reagent cross-contamination. Label three 12.5 mL tips "Diluent", "Substrate" and "Stop," and one 5.0 mL tip "Conjugate".
• Draw the desired reagent volume into the Repeater pipettor and dispense one portion of the reagent back into the container to properly engage the ratchet mechanism. If you do not do this, the first volume delivered may be inaccurate.
• To add reagents using the Repeater pipettor, pipette down the side of the test tube just below the rim.
• To add samples and calibrators using the positive displacement pipettor, pipette down the side of the test tube just above the liquid level.
• The carryover volume of the positive displacement tips is minimal, but may affect results if you are going from a high to low DDT concentration. Use a new pipettor tip each time you pipette a new unknown.

Assay Procedure

Collect/Store the Sample

1. Collect soil in appropriately sized and labeled containers.
2. Take care to remove excess twigs, organic matter and rocks or pebbles from the sample. For best results, wet soils should be air-dried overnight and thoroughly mixed before testing.
3. Store soil samples at 4°C (39°F).

Prepare the Sample/Extract the Soil

1. Please follow the instructions from the EnviroGard Soil Extraction Bottle Kit to prepare the soil extract before the assay.
2. **5 ml** of **Methanol** will be used to extract DDT residue from a 5 gram soil sample. As per instructions, attach a **50 mL** Combitip to the Repeater pipettor and set the dial to **5**. Deliver once to add **5 mL** of **methanol** to the extraction vial, and cap tightly.

Perform the Test

NOTE: Allow all reagents and sample extracts to reach room temperature before you begin the test. Do not analyze more than 20 test tubes at a time.

1. The choice of calibrators to use in the test will depend on the selection of the analyst. The use of two calibrators may be appropriate if screening for a single level of DDT.

Remove the test tubes from the plastic bag and label them as follows*:

Tube Label	Tube Contents
NC	Negative Control
C1	0.2 ppm Calibrator
C2	1.0 ppm Calibrator
C3	10.0 ppm Calibrator
S1	sample 1
S2	sample 2
etc.	

You are not required to perform the assay in duplicate; however, doing so will increase the precision.
Place the test tubes in the test tube rack. Push down on each tube so that it is held firmly and does not fall out of the rack when shaken.
CAUTION: Do not "snap" the test tubes into the rack as this may result in a cracked tube.

2. Attach the **12.5 mL** Combitip labeled "Diluent" to the Repeater pipettor and adjust the dial to **2**. Add 500 microliters (µL) of Assay Diluent to each test tube.
3. Attach a clean pipette tip to the Microman pipettor and adjust the dial to "250". Add 25 µL of each calibrator (including Negative Control) to the corresponding test tube by placing the end of the pipette tip against the side of the tube (just above the level of the Assay Diluent) and dispensing the volume. Use a clean pipette tip each time.

CAUTION: Replace the caps on the calibrator vials immediately after use to minimize evaporation.
4. Using a clean tip for each sample, add 25 µL of each sample extract to the appropriately labeled test tube.
5. Attach the **5.0 mL** Combitip labeled "Conjugate" to the Repeater pipettor and adjust the dial to **1.** Add 100 µL of DDTEnzyme Conjugate to each test tube.
6. Shake the test tube rack to mix for 10 to 15 seconds. Leave the test tubes undisturbed for 15 minutes.
7. Vigorously shake out the test tube contents into a sink or suitable container. Fill the test tubes to **overflowing** with cool tap or distilled water, then decant and vigorously shake out the remaining water.
Repeat this wash step three more times, being certain to shake out as much water as possible on each wash. After the final wash, remove as much water as possible by tapping the inverted tubes on absorbent paper.
8. Attach the **12.5 mL** Combitip labeled "Substrate" to the Repeater pipettor and set the dial to **2**. Add 500 µL of Substrate to each test tube. Leave the test tubes undisturbed for 10 minutes.

NOTE: If a blue color does not develop in the Negative Control test tube within 10 minutes after adding the Substrate, the test is invalid and you must repeat it.

Interpret the Results

You can either interpret the results visually within 10 minutes after adding the Substrate to each test tube, or you can perform a more precise analysis with a photometer after you add the Stop Solution.

Visual Interpretation

After you add the Substrate, wait 10 minutes then mix the test tubes by shaking them for a few seconds until they are a uniform blue color. Compare the sample test tube to the calibrator test tubes against a white background. The test tube rack in the kit is well-suited for this purpose.

NOTE: The word DDT in the interpretation instructions below refers to "total DDT", i.e. the sum of p,p'-DDT, p,p'-DDD, and p,p'-DDE.

• If a sample test tube contains more color than the calibrator test tube, the sample contains DDT at a concentration lower than the calibrator.
• If a sample test tube contains less color than the calibrator test tube, the sample may contain DDT at a concentration greater than the calibrator.
• If the sample test tube contains color that is between the calibrator test tubes, the sample contains DDT at a concentration between the calibrator concentrations.
• If a sample test tube contains approximately the same amount of color as the calibrator test tube, the sample contains DDT at a concentration approximately equal to the calibrator.
• If the sample test tube contains less color than the 10 ppm Calibrator test tube, you may dilute a fraction of the soil extract in methanol (for example, 1:100) and perform the assay again. To determine the

concentration of the diluted extract multiply the result by the dilution factor. (Go to "Semi-Quantitative Interpretation" for further details.)

Photometric Interpretation

After you add the Substrate, wait 10 minutes then add the Stop Solution to each test tube.
WARNING: Stop solution is 1N Hydrochloric acid.

Attach the **12.5 mL** Combitip labeled "Stop" to the Repeater pipettor and set the dial to **2**. Add 500 µL of Stop Solution to each test tube. This converts the blue color in the test tubes to yellow.

NOTE: After you add Stop Solution to the test tubes, results should be read within 30 minutes.

Differential Photometer

1. Place a water blank test tube containing 1.5 mL of deionized water, or equivalent in the left (reference) well.
2. Place the Negative Control test tube into the right (sample) well. Record the optical density (OD) of the Negative Control.
3. Remove the Negative Control test tube and replace it with the 0.2 ppm Calibrator test tube to reactivate the photometer. Record the result. Repeat this step to determine the OD for each of the remaining calibrators and for each sample.

Semi-quantitative Interpretation

Compare the OD of each sample to the OD of each calibrator:
NOTE: The word DDT in the interpretation instructions below refers to "total DDT", i.e. the sum of p,p'-DDT, p,p'-DDD, and p,p'-DDE.

- If a sample OD is equal to the OD of a calibrator, the sample contains DDT at a concentration approximately equal to the calibrator.
- If a sample OD is greater than a calibrator OD, the sample contains less DDT than the calibrator.
- If a sample OD is lower than a calibrator OD, the sample may contain more DDT than that calibrator.
- If an assay result indicates that a soil sample contains greater than 10 ppm total DDT, but you need more specific information, the soil extract may be diluted 1:100 in neat methanol, and assayed again. You must then multiply the results of the re-assay by 100 to determine the approximate sample concentration.

NOTE: If you know in advance that the "action level" of interest is greater than 10 ppm total DDT in soil, the assay may be modified to pinpoint that particular concentration. For example:

If you wish to categorize samples as less than or greater than 250 ppm, you should dilute all sample extracts 1:250 in neat methanol (e.g. 20 µL extract plus 4.98 mL methanol) and compare the diluted extracts to the 1 ppm DDT kit calibrator. Due to the 250-fold dilution, the 1 ppm calibrator represents 250 ppm in the assay.

NOTE: If you are interested in action levels greater than 1000 ppm, please contact Technical Assistance for assistance.

Limitations of the Procedure

The EnviroGard DDT in Soil Test Kit is a qualitative/semi-quantitative screening test only. Actual quantitation of DDT by EnviroGard immunoassay is not possible due to the Test kit's cross-reactivity with DDT breakdown products and other similar compounds and to the variations in extraction efficiency inherent in the fast extraction protocol described in this product insert.

Soil sampling error may significantly affect testing reliability. The distribution of pesticides in different soils can be extremely heterogeneous. Soils should be dried and homogenized before analysis by any method. Split samples (i.e. for GC and immunoassay) should always derive from the same homogenate.

Ordering Information

Description	Catalog Number
EnviroGard DDT in Soil Test Kit	73100
EnviroGard Soil Extraction Bottle Kit	72010

Technical Assistance

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General Limited Warranty

Strategic Diagnostics Inc. warrants the products manufactured by it against defects in materials and workmanship when used in accordance with the applicable instructions for a period not to extend beyond a product's printed expiration date. **SDI MAKES NO OTHER WARRANTY, EXPRESSED OR IMPLIED. THERE IS NO WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE.**

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